

OPEN LOOP DYNAMICS OF A DISTILLATION COLUMN

**A Thesis Submitted
In Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY**

**By
S. DIKSHITULU**

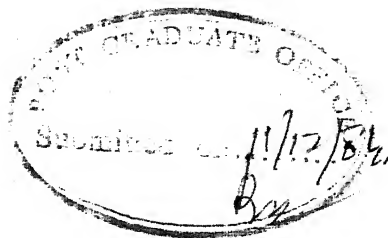
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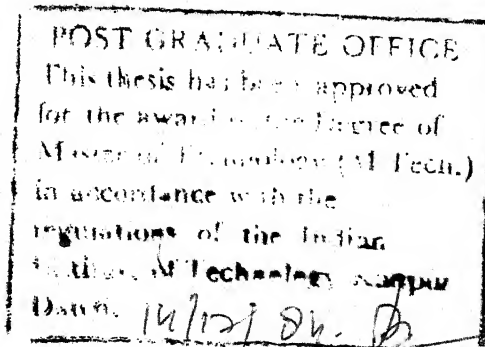
It is certified that the present work entitled "OPEN LOOP DYNAMICS OF A DISTILLATION COLUMN" has been carried out by Mr. S. Dikshitulu under my supervision and that it has not been submitted elsewhere for a degree.

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Acknowledgements

I am thankful to Dr. D.N. Saraf for providing discerning guidance, valuable advice and constructive criticism throughout the course of this investigation. Thanks are due to Mr. A. Khanna for providing valuable suggestions from time to time.

I also thank Mr. Samarjeet Singh, Technical Officer, for having helped me with the experimental part of this work.

Thanks are due to Mr. Jacob John and Mr. V. Manuja for giving assistance in the instrumentation.

I thank Mr. Shivdayal Singh, Mr. Raj Kumar, Mr. Ayodhya Singh and Mr. Kalicharan of Unit Operations Lab. for assisting me during the experimental runs.

I am thankful to my friends Mr. V.L.N. Murthy and Mr.V.S. Gangadhar Rao for having helped me in this investigation and who made my stay at I.I.T. Kanpur cheerful.

Thanks are due to Mr. B.S. Pandey for typing this thesis really at a very short notice and to Mr. A.K. Ganguly for the drawings.

S. Dikshitulu
Author

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NOMENCLATURE

- a_n = parameter as defined in text (Table 1)
 B = bottom product flow rate, moles/minute
 b_n = parameter as defined in text, (Table 1)
 D = top product flow rate, moles/minutes
 E = Murphree vapor plate efficiency
 f = perturbation in feed rate, moles/minute
 F = feed rate, moles/minute
 G_{nu} = gain for plate n composition response to a load u :
 G_{nx} to feed composition, G_{nF} to feed rate
 H = holdup moles; H_T is total column holdup
 K_n = constant as defined in text (Eqn.15)
 L_R = reduced circulation rate
 L = liquid flow rate, moles/minute
 N = tray number
 R = reflux ratio
 T = time constant; T_z is reciprocal zero, T_1, T_2, T_3, T_4 are first, second, third and fourth time constants respectively, minutes
 T_s = simplified model time constant, minutes
 u = load variable, if feed rate, x_F is feed composition
 x = perturbation in composition of acetone, mole fraction
 X = mole fraction of acetone in liquid phase
 y = perturbation in vapor composition of acetone, mole fraction

Y = mole fraction of acetone in vapor phase

Greek letters

= relative volatility

= constant as defined in text (Table 1)

Subscripts

A = acetone

B = bottom product

D = top product

F = feed

n = tray location, 0 is condenser,
N+1 is reboiler

R = Reboiler

W = Water

Superscripts

1 = first steady state

2 = second steady state

ABSTRACT

The open loop dynamics of a pilot scale distillation column was studied as part of a large project envisaging direct digital control for the process. The variables that affect the steady state operation of a distillation column are: feed flow rate, feed composition, reflux flow rate, reboiler steam pressure and feed temperature. The transfer functions for the distillation column were obtained for step disturbances in feed flow rate and feed composition using a model proposed by Wahl and Harriot. This was a preliminary investigation and there were numerous practical problems encountered during the column operation primarily because of lack of control. For change in feed flow rate, the proposed model predicted the steady state behavior fairly well but the experimental results during unsteady operation did not match with the prediction mainly because of the assumption of constant feed rate before and after the disturbance was violated. For feed composition disturbance, the model did not agree with the observations at all because of the large step size which resulted in significant change in molar feed flow rate contrary to the assumptions.

CHAPTER 1

INTRODUCTION

Dynamic modelling is essential to study the unsteady behaviour of a process. The increased concern about the control of industrial processes has led to a pronounced interest in the study of their dynamics. Distillation is one of the most widely used processes in chemical industry. It requires a good amount of energy for its operation and often stringent duty is assigned to it and therefore its control within close limits is essential for an economical and efficient running of the plant.

Models for process dynamics may be obtained theoretically or experimentally. A theoretical analysis is based on the application of the basic physical laws governing the process. However, these models tend to get very cumbersome computationally and may require extensive experimentation for evaluation of the parameters involved. In a purely experimental approach, the process is considered as a black box, and, the output is related empirically to the changes in input variables. This gives the dynamic behavior but does not allow an insight into the process, as the basic physical parameters will not appear in the transfer function explicitly. A more realistic model is usually a combination of the two where, based on theoretical

knowledge one chooses a model and simplifies it in the light of the knowledge of the experimental observations and finally a few parameters involved are evaluated empirically.

Distillation dynamics can be expressed, quite simply even for multicomponent distillation in terms of state variables (Toijala and Gustaffson, 1971) by writing unsteady state material and energy balance equations. However, such a model is difficult to work with, in columns having many equilibrium stages. The study of control systems through state variable models for such columns would require large storage space and computing time. A theoretical model in all its detail is not very suitable, for the control of a process since the amount of computational effort is large. The complexity of the process model has to be reduced, also because it is likely to contain unnecessary information. Toijala (1972) and Astrom and Eykhoff (1971) used process identifiical methods to obtain simple models for complex systems. In process modelling for control, the degree of sophistication of the model is a function of the control theory used where usually, only the basic structure of the process is needed. Therefore, simpler models are more desirable.

In an operating distillation column the variables which may experience random disturbances and which need to be controlled are:

- 1) Feed flow rate
- 2) Feed composition
- 3) Reflux flow rate
- 4) Vapor flow rate
- 5) Feed temperature

However, in order to extract an appropriate model, planned disturbances can be given. These may be

- 1) Step disturbance
- 2) Pulse disturbance
- 3) Sinusoidal disturbance

In this investigation, the composition response of a multistage pilot scale distillation column was studied for step changes in feed flow rate and feed composition. This is a part of a continuing project envisaged to provide digital control for the column. While, for control, it is necessary to study closed loop dynamics, the present study is preliminary, and only the open loop dynamic response of the column was investigated.

A survey of various distillation column models is given in Chapter 2, along with a description of the model chosen for this investigation. Chapter 3 discusses the experimental setup and operation of the distillation column. The results and discussions of the experimental runs are given in Chapter 4.

Modifications in the experimental setup are suggested here for a smoother operation of the column. In the last Chapter, concluding remarks alongwith suggestions for future work are included.

CHAPTER 2

DISTILLATION COLUMN MODELS

Process modelling from first principles usually results in very complicated models. On the other hand, a theoretical model with simplifying assumptions, and requiring some parameters to be found experimentally, usually can represent a fairly complicated process in a simple form. This is because a number of small effects, combine into a few larger overall effects (Astrom and Eykhoff, 1971). The correlations should be made such that as many of the limiting assumptions are absorbed in the parameters are possible, to increase the model accuracy. The simplest case would be that of a one parameter model. For a constant holdup and overflow rate, Robinson and Gilliland (1950) have shown that the response to a feed composition change in terms of a single time constant. Another such simple model is that of Armstrong and Wilkinson (1957). The accuracy in these models is not high, but they can be used for quick calculations to obtain order of magnitudes. For changes in feed composition and reflux flow rate, the response of the reboiler and the condenser has been reported by Williams et al. (1965, 1972). The composition response of all the stages, to all of the commonly encountered disturbances, has been shown by Wahl and Harriot (1970), Waller (1968, 1969, 1970, 1971), and Waller

and Jonasson (1970, 1972). In both these models the flow dynamics was neglected and the composition dynamics studied. The two principal parameters in Waller's model are obtained from the number of real plates, the holdups, the flows and the plate gains, i.e. from steady state data only. These parameters, T and P, are defined for the enriching section (e) and stripping section (s) as follows:

$$T_e = \frac{(M+2) H_{eq}^0}{F^0} \quad (1a)$$

$$T_s = \frac{(N+1) H_{eq}^0}{F^0} \quad (1b)$$

where

M = number of real plates in enriching section

N = Number of real plates in stripping section

F^0 = feed flow rate at steady state, Moles/sec

$H_{eq}^0 = (M+N+2)H_p$, total hold up, moles

H_p is hold up per plate.

$$P_e = \sqrt{\frac{10}{M+2}} \times Q \sqrt{5 \frac{10}{M+2}} \quad (2a)$$

$$P_s = \sqrt{\frac{10}{N+1}} \times Q \sqrt{5 \frac{10}{N+1}} \quad (2b)$$

where Q is defined as

$$Q = K_{eq} \left(\frac{V_s^0}{F^0} + 1 \right) \quad \text{for } q^0 > 0.5 \quad (3a)$$

$$Q = K_{eq} \left(\frac{L_e^0}{F^0} + 1 \right) \quad \text{for } q^0 < 0.5 \quad (3b)$$

with

$$K_{eq} = \frac{\sum_{j=0}^{M+N+1} K_j / (M+N+2)}{\frac{D^0}{F^0} K_o + \frac{B^0}{F^0} K_R} \quad (4)$$

where

K_j = gain on plate j , $j=0$ is condenser and
 $j = M+N+1$ is reboiler

V_s = vapor flow rate in stripping section, moles/sec

L_e = liquid flow rate in enriching section, moles/sec

The response curves for equal holdup in plates, condenser and reboiler, are given in (Waller, 1978).

The model suggested by Wahl and Harriot (1970) considers the differential equations for component material balances. The four points where disturbances can enter this process are

- 1) the feed composition, x_F
- 2) the feed flow rate, f
- 3) the boilup rate, v
- 4) the reflux rate, r

Change in the temperature of the feed corresponds to altering the reflux and vapor rate at the feed plate, and so is a

special case of 3) and 4) applied to a part of the column and therefore is not listed separately.

The dynamic response can be expressed as

$$x_n(t) = \mathcal{L}^{-1} \left[x_f(s) \left(\frac{x_n(s)}{x_f(s)} \right) + f(s) \left(\frac{x_n(s)}{f(s)} \right) + v(s) \left(\frac{x_n(s)}{v(s)} \right) + r(s) \left(\frac{x_n(s)}{r(s)} \right) \right] \quad (5)$$

where

$x_n(t)$ = perturbation in composition of plate n at time t

$x_f(s)$ = feed composition perturbation in Laplace domain

$f(s)$ = feed rate perturbation in Laplace domain

$v(s)$ = vapor rate perturbation in Laplace domain

$r(s)$ = reflux rate perturbation in Laplace domain

$\frac{x_n(s)}{x_f(s)}$, $\frac{x_n(s)}{f(s)}$, $\frac{x_n(s)}{v(s)}$ and $\frac{x_n(s)}{r(s)}$ are the transfer functions for each of the disturbances.

A component material balance in the column gives the following relation between the input and output quantities for each of the four loads (Figure 1). The assumption made is that the column is considered as a black box. The black box contains the total amount of material (liquid and vapor holdups) which is about constant, but the average composition (x_{av}) which varies.

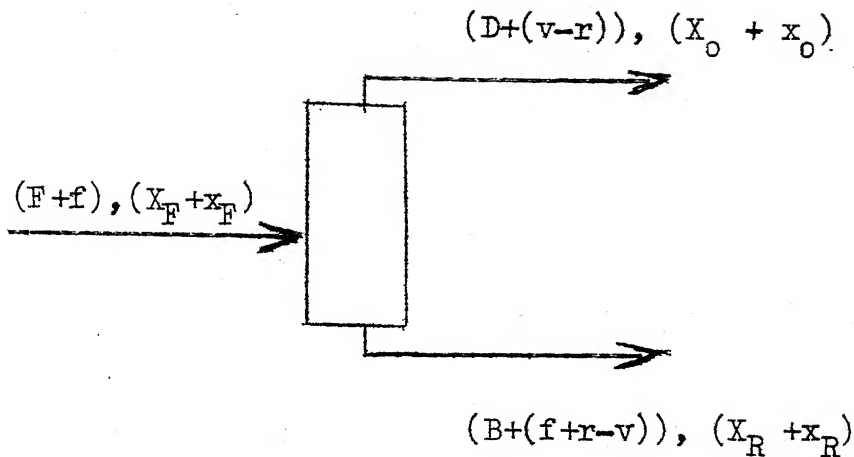


Figure 1: Distillation Column as a Black Box

$$H_T \frac{dx_{av}}{dt} = - Dx_O - Bx_R + Fx_F \quad (6)$$

$$H_T \frac{dx_{av}}{dt} = - Dx_O - Bx_R + f(X_F - X_R) \quad (7)$$

$$H_T \frac{dx_{av}}{dt} = - Dx_O - Bx_R + v(X_R - X_O) \quad (8)$$

$$H_T \frac{dx_{av}}{dt} = - Dx_O - Bx_R + r(X_O - X_R) \quad (9)$$

(For detailed derivation see Appendix 1)

where

H_T = total liquid holdup, moles

F = feed flow rate, moles/min

B = bottoms flow rate, moles/min

D = distillate flow rate, moles/min

X_F = feed composition, mole fraction

X_O = distillate composition, mole fraction

X_R = bottoms composition, mole fraction

The interior of the column is described as a set of $N+2$ well mixed tanks which are maintained at steady state, and are upset by loads introduced into this system at some point and withdrawn at the others. The circulating flow consists of the vapor stream in one direction and the liquid stream in the other.

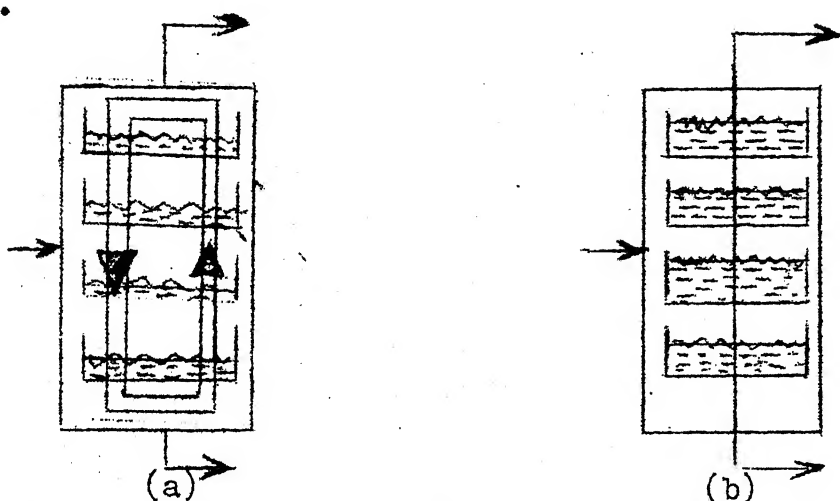


Figure 2: Essential Interior of a Distillation Column

(a) low holdup, high circulation rate

(b) High holdup, low circulation rate

The parameter which describes the ability of the system to maintain itself at equilibrium is the ratio of the circulation rate to the holdup (Figure 2). The reciprocal of this can be considered as a mixing or circulation time of the column. It is a capacitance, H_T , divided by the flow rate, L through the capacitance. On the other hand, the rate at which the system responds to the upsets will be given by a characteristic time

constant for the system, T_s . T_s is approximately equal to the principal time constant of the system. Because of the importance of these two effects, the ratio of the two is taken as reduced circulation rate, L_R .

$$L_R = \frac{(L/H_T)}{(1/T_s)} = \frac{T_s L}{H_T} \quad (10)$$

Linearizing and solving each of equations (6) to (9) results in solution of T_s as

$$T_s = \frac{\sum_{n=0}^{N+1} H_n G_{nu}}{B G_{RU} + D G_{OU}} \quad (11)$$

where

G_{nu} = Gain on plate n due to load change u , u is x_F or f or v or r

$n = 0$ is condenser and $n = N+1$ is the reboiler

(For detailed derivation of equation (11) see Appendix 1).

Transfer Functions:

The dynamic terms in the transfer functions for the column may be estimated from the two parameters T_s and L_R and other basic physical parameters of the column. The exact transfer functions for the column for perturbation loads were found by Wahl and Harriot who solved over 300 cases of binary distillation columns. They found that the time constants for all the plates for all types of loads are identical and the differences that occur are because of variations in the values of zeros.

Except for four time constants T_1 , T_2 , T_3 and T_4 and one zero T_z , all other time constants were found to be negligible. For feed composition load, adequate representation was obtained in the time domain by using the principal time constant and the third largest time constant, providing the proper zero is used. The first and the third largest time constants were used because the second and fourth were effectively cancelled by the zeros. For a feed rate load, however, the first and the second time constants should be used for top of the column, since the third and not the second time constant was effectively cancelled by a zero. For the bottom of the column, only the principal time constant was used, as the remaining time constants were effectively cancelled by the zeros. Correlation graphs for estimating these time constants are given in Appendix 2.

The suggested transfer functions are as follows:

$$x_F(s) \rightarrow \boxed{\frac{G_{nx}(T_z s + 1)}{(T_1 s + 1)(T_3 s + 1)}} \rightarrow x_n(s) \quad (12)$$

For bottom of column

$$f(s) \longrightarrow \boxed{\frac{G_{nf}}{(T_1 s + 1)}} \longrightarrow x_n(s) \quad (13)$$

For top of column

$$f(s) \longrightarrow \boxed{\frac{G_{nf} (T_z s + 1)}{(T_1 s + 1) (T_2 s + 1)}} \longrightarrow x_n(s) \quad (14)$$

The gains, G_{nu} , can be calculated from the steady state compositions for two values of any load parameter using the equations given in Table 1. The derivations are given in Appendix 1. This procedure requires the value of the change in vapor composition with change in liquid composition on each plate, K_n .

$$K_n = \left(\frac{dy}{dx} \right)_n \quad (15)$$

K_n can be obtained from equation (16) for constant relative volatility and constant plate efficiency.

$$K_n = \frac{E \alpha}{(1 + (\alpha - 1)x)^2} + (1-E)\left(\frac{I}{V}\right) \quad (16)$$

The equations in Table 1 can be used if there is a total condenser, equimolar overflow, a single feed stream, and top and bottom product streams only. These restrictions can be relaxed by following the procedure given in Appendix 1.

TABLE 1: CALCULATION OF PLATE COMPOSITION GAINS

Step 1. Determination of a_n and b_n

n	b_n	Common term \mathcal{E}
0	1	0
1	$1/K_1$	0
$2, \dots, N_F$	$\left[\frac{V_e K_{n-1} + L_e}{V_e K_n} \right] b_{n-1} - \frac{L_e}{V_e K_n} b_{n-2}$	$\left[\frac{V_e K_{n-1} + L_e}{V_e K_n} \right] a_{n-1} - \left[\frac{L_e}{V_e K_n} \right] a_{n-2}$
$N_F + 1$	$\left[\frac{V_e K_{n-1} + L_e}{K_n V_s} \right] b_{n-1} - \frac{L_e}{V_s K_n} b_{n-2}$	$\left[\frac{V_e K_{n-1} + L_e}{V_s K_n} \right] a_{n-1} - \left[\frac{L_e}{V_s K_n} \right] a_{n-2}$
$N_F + 2 \dots N_R$	$\left[\frac{V_s K_{n-1} + L_s}{K_n V_s} \right] b_{n-1} - \frac{L_s}{V_s K_n} b_{n-2}$	$\left[\frac{V_s K_{n-1} + L_s}{K_n V_s} \right] a_{n-1} - \left[\frac{L_s}{V_s K_n} \right] a_{n-2}$

 a_n for load shown

n	Feed composition	Feed rate
0	0	0
1	0	0
$2, \dots, N_F$	0	0
$N_F + 1$	$\frac{-F}{V_s K_n}$	$\mathcal{E} + \frac{X_{n-1} - X_F}{V_s K_n}$
$N_F + 2 \dots N_R$	\mathcal{E}	$\mathcal{E} + \frac{X_{n-1} - X_F}{V_s K_n}$

Table 1 contd.

Step 2: Determination of Condenser gain G_{ou}

$$\text{For feed composition load } G_{ox} = \frac{F - Da_R}{D + Bb_R}$$

$$\text{For feed rate load } G_{oF} = \frac{X_F - X_R - Ba_R}{D + Bb_R}$$

Step 3: Determination of gain on the plates

For all loads: $G_{nu} = a_n + b_n G_{ou}$. b_n and a_n for appropriate loads are given above.

The distillation column was simulated using the model suggested by Wahl and Harriot. The model suggested by Waller could also have been used, but it has a further simplifying assumption that the holdup on all the plates, the reboiler and condenser are same. But for this difference both the models are quite similar. A comparison between the two models has not revealed any significant differences concerning the amount of work needed or accuracy obtained (Waller 1978).

In both these models, the disturbances are considered independently. They represent models for single input - single output system. The responses to all the disturbances can be obtained by superposition as shown in equation (5). This will give a representation of multiple input-single output system. For multiple input-multiple output system the distillation column can be modelled by (Meyer et al. 1979 and Oguniake and Ray, 1979).

$$\underline{Y}(s) = \underline{G}(s) \underline{U}(s) + \underline{G_d}(s) \underline{d}(s) \quad (17)$$

where

$\underline{Y}(s)$ = vector of output variables in Laplace domain,
such as overhead composition, side stream
composition etc.

$\underline{U}(s)$ = vector of input or control variables such as
reflux flow rate, reboiler steam pressure etc.

$\underline{d}(s)$ = vector of disturbance variables in Laplace

domain such as feed composition, feed flow rate.

The elements of the matrices $\underline{G}(s)$ and $\underline{G}_d(s)$ can be found by keeping all except one, of the elements of $\underline{U}(s)$ and $\underline{d}(s)$ zero. The effect of that variable on the output can then be studied. These models have time delays incorporated in them. These models though more complex are a better representation of the distillation column. But, this requires an on-line sample analyzer in order to measure the time delays accurately. Since an on-line sample analyzer was not available, this model could not be used in the present study.

Once the transfer functions have been obtained, the model can be used to study control loop strategies (Beaverstock and Harriot, 1973). To control top product by reflux manipulation the best location of the sensor would be the top product itself. (Wahl and Harriot, 1971) as the steady state error is minimized. For step changes in load, the optimum control sensor location is plate 1. For a multiple input-multiple output distillation column, various control strategies have been suggested by Smith (1957), Meyer et al. (1979), Ogunnaike (1980) and Rosenbrock (1974).

CHAPTER 3

EXPERIMENTAL WORK

3.1 Experimental Setup:

The pilot scale, multistage distillation column has been designed as a general purpose tower which can handle common systems such as methanol-water, ethanol-water, water-acetic acid, benzene-toluene, acetone-water etc. The design is based on the average properties of the compounds mentioned above. It can also be used to distill multicomponent mixtures of some of the above compounds.

It is a sieve plate column made of stainless steel, with the following specifications:

i) Number of stages	16
ii) Column diameter	22.86 cm
iii) Stage height	22.86 cm
iv) Thickness of tray	0.32 cm
v) Weir length	16.76 cm
vi) Number of holes/tray	140
vii) Spacing	Triangular
viii) Diameter of holes	0.635 cm
ix) Pitch	1.52 cm.

A thermometer pocket has been welded to each stage for insertion of platinum resistance thermometer. The thermometer

is so fixed as to locate the sensor within 3 mm above the sieve plate in order to sense the liquid temperature on the tray. Also every stage has been provided with 6 mm sample outlet pipe, welded near the bottom flange about 25 mm above the tray. Trays 4 to 12 have been provided with feed inlet pipes of 2.54 cm diameter. A steel skeleton framework provides support for the column. The bottoms accumulator has been mounted on a brick foundation and the stages assembled on it.

Reboiler Specifications:

It is a vertical thermosiphon type of reboiler, and has been designed for a heat duty of 64×10^6 cal/hr. It has 31 tubes of stainless steel. The shell is made of mild steel but has been electroplated from inside with nickel and chromium:

Number of tubes	31
O.D. of tubes	18 mm
Length of tubes	60 cm
Pitch	30 mm
Spacing	triangular
BWG of tubes	16

Condenser Specifications:

The condenser is a 1-2 shell and tube type. It has been designed for a heat duty of 37.5×10^6 cal/hr.

Number of tubes	59
O.D. of tubes	19 mm

Length of tube	60 cm
BWG of tube	16
Tube material	Stainless steel
Baffles material	Mild steel
Shell material	Mild steel

The shell was electroplated inside with Nickel and Chromium.

Equipment Layout:

The distillation column is located in the Unit Operations Laboratory. The location of accessories is as follows:

- | | |
|---------------|------------------------|
| First Floor: | 1) Reboiler |
| | 2) Bottom product drum |
| Second Floor: | 1) Feed preheater |
| | 2) Instrument panel |
| Third Floor: | 1) Feed drum |
| | 2) Condenser |
| | 3) Reflux accumulator |

The column and the reboiler have been insulated with glass-wool and lagged with magnesia. There are no controllers in the setup which make the model tedious and laborious. The general layout is given in Figure 3.

Measurement of Temperature:

Temperature is measured using platinum resistance thermometers. (Instrumentation Limited, Kota). The specifications are given below:

Temperature range	0° to 500°C
Thermometer element	Platinum
Thermometer resistance	100 at 0°C
	140 at 100 °C
Maximum operating pressure	40 kg/cm ²
Working length	1250 mm
Overall length	1410 mm
Type	PRT 101

The principle used here is that resistance of certain materials, such as platinum is very sensitive to temperature. For pure materials, the change in resistance is practically linear over a certain range. The change in resistance is transduced into voltage signals. The voltage signal is then amplified to the required range. The resistance vs temperature graph and the associated voltage conversion circuit are shown in Appendix 3.

Control Valves:

Four control valves, pneumatic type (Instrumentation Limited, Kota) have been placed to control the feed flow, reflux flow, bottoms flow and the steam flow. The operating air pressure is 3 to 15 psi.

Flow Measurement:

There are four orifice plates, one in the steam line (1.27 cms diameter) and one each in reflux, feed and bottom (0.635 cms diameter) line. The pressure drop across the orifice plates can be measured using a differential pressure (DP) cell which, in turn, can be used to calculate the flow rates. The appropriate reflux ratio, the maximum column capacity, the reboiler and the condenser duty are shown in Appendix 3. Semi conductor type DP cells (National semiconductors) were used to measure the pressure drop across the orifice plates. The chip housing was filled with silicone oil to prevent the working fluid from coming in contact with the cell. The silicone oil was separated from working fluid by using a latex rubber sock which allowed pressure to be transmitted but did not allow fluid mixing. However, the sock tended to rupture with prolonged usage and at times flow had to be measured directly by collecting the flow for a fixed time.

Composition Measurement:

In binary distillation it is possible to relate the composition of a tray to its temperature through a T-X,Y diagram and hence monitoring temperature of each tray is equivalent to monitoring its composition. However, as a check, samples of liquid holdup were collected periodically from sample ports in 30 ml. Stoppered sampling bottles and composition checked

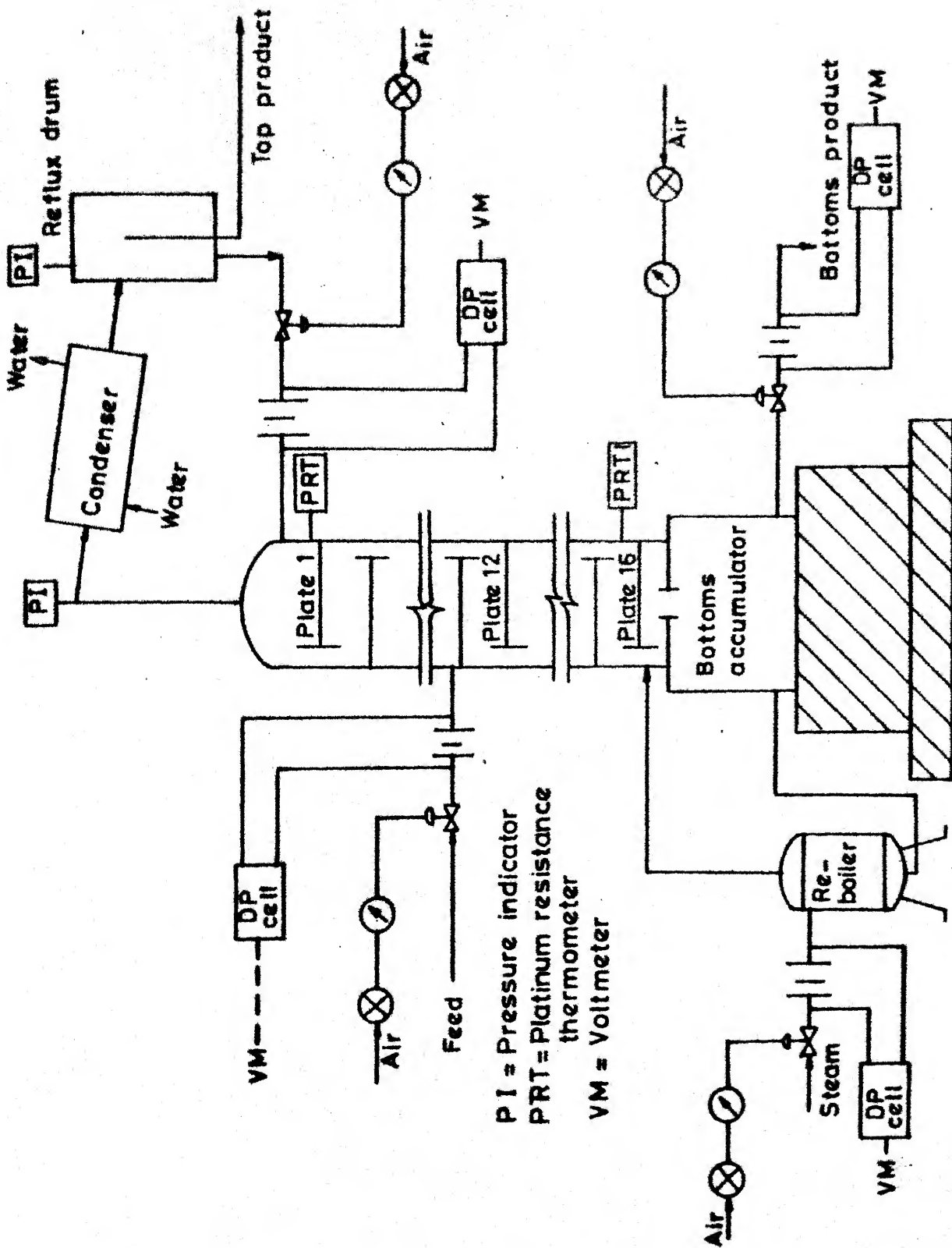


FIG. 3 Distillation column

by measuring the refractive index with an Abbe Refractometer

This process was used only to verify the composition at selected positions as obtained from temperature measurements.

3.2 Operating Procedure:

Feed consisting of a known mole percent of acetone in water was introduced at a predetermined flow rate into the column. This was done after preheating the column for about an hour by supplying steam to the reboiler. Feed was introduced at the twelfth tray. The condenser water supply was turned on, the moment the feed was introduced. The column was operated under total reflux, for approximately an hour, to enable the holdup in the enriching section to build up fast, after which it is operated at finite reflux. The bottom product valve was opened, when the feed was introduced. The feed flow rate, the bottoms flow rate and the reflux flow rate were adjusted manually using the pneumatic control valve. This was done keeping the steady state values (for details see Appendix 3).

Samples are drawn periodically from the top product, bottom product and trays 1, 10 and 16. The composition was analyzed using an Abbe Refractometer. The temperatures on all the trays were also measured periodically. The bottoms product was collected in the bottoms tank. Excess of the top product from the reflux accumulator also drained into the bottom product tank.

The mixture (top and bottom products) collected in the bottoms drum must have the same composition as the feed. This is recycled periodically using a centrifugal pump.

After steady state operation was achieved, i.e. when the temperatures became constant, a step change was given in feed rate/feed composition. The temperatures and the compositions were again measured till the second steady state was achieved. The feed supply to the column was stopped along with the steam to the reboiler after the second steady state was achieved. After closing the bottom product valve, the total column holdup was measured manually.

CHAPTER 4

RESULTS AND DISCUSSIONS

The model proposed by Wahl and Harriot (1971) and discussed earlier was used to simulate the present distillation column because of its simplicity. The following assumptions were made:

1. Volumetric liquid holdup on all trays is equal.
2. Vapor holdup is negligible compared to liquid holdup.
3. Equimolar overflows of vapor and liquid hold (McCabe Thiele assumption).
4. Temperature measurement at any tray can be used to calculate composition using T-X,Y diagram.
5. Reflux temperature was at bubble point. This assumption was not strictly correct since reflux was invariably cold because of lack of temperature control.
6. Plate efficiency is constant ($E=0.75$).

The following experimental runs were undertaken, two each for feed rate and feed composition change. The operating conditions for each of the runs is listed in Table 2.

TABLE 2: OPERATING CONDITIONS

Run No.	Disturbance Variable	Step Size	Initial Parameters			Remarks
			Feed rate, F $\frac{\text{gm}}{\text{min}}$	Feed comp., X_F mole fract.	Steam press., P kg/cm^2	Reflux ratio
1	Feed rate	12.5%	35.38	0.15	2.2	2.2
2	Feed composition	16.6%	35.38	0.15	2.2	2.2
3	Feed rate	12.5%	35.38	0.15	2.2	2.2
4	Feed composition	-46.6%	35.38	0.15	2.2	2.2

Run no.2
abandoned
on account
of vapor
locking in
feed line

TABLE 3: GAINS FOR ALL THE PLATES

Plate Number	Run 1	Run 3	Run 4
Condenser	0.0222	5.5847	0.0251
1	0.0046	0.9548	0.0058
2	0.0021	0.4363	0.0026
3	0.0017	0.3631	0.0022
4	0.0017	0.3384	0.0022
5	0.0017	0.3165	0.0024
6	0.0017	0.2470	0.0026
7	0.0017	0.1449	0.0029
8	0.0017	0.2228	0.0047
9	0.0017	0.0113	0.0156
10	0.0024	0.0582	0.0119
11	0.0025	0.2690	0.0039
12	0.0031	0.2511	0.0021
13	0.0032	0.0284	0.0019
14	0.0023	-0.0464	0.0015
15	0.0016	-0.0721	0.0010
16	0.0010	-0.0907	0.0006
Reboiler	0.0004	-0.0979	0.0002

TABLE 4: TIME CONSTANTS FOR FOUR TYPICAL TRAYS

Plate Number	T_1 (min)	T_2 (min)	T_3 (min)	T_4 (min)	T_z (min)
1	47.66	18.05	12.70	10.16	1.124
11	47.66	18.05	37.15	29.72	37.16
13	47.66	18.05	33.73	26.98	-6.72
16	47.66	18.05	38.38	30.70	-11.20

Because of enormous experimental difficulties, to be discussed later, it was not possible to get more extensive data in the limited time available.

Measured values of the bottoms and distillate flow rate, holdups and the calculated values of the gains were used to calculate T_g using equation (11). The gain for each plate was calculated using equations in Table 1 and are given in Table 3. The details of determining the reflux ratio as well as molar flow rates are given in Appendix 3. The time constants were found by correlations given by Wahl and Harriot. These correlation curves are given in Appendix 2 for convenience. The time constants found for four typical trays are given in Table 4. Equations 12 to 15 were then used to obtain transfer functions for various plates as obtained below. Two plates below the feed and two above were chosen to demonstrate the column behavior. Plates 1 and 11 are above the feed and 13 and 16 are below it.

The transfer functions are as follows:

For feed rate load, f

$$\text{Plate 1: } \frac{x_n(s)}{f(s)} = \frac{.0058 (1.124 s+1)}{(47.66s+1)(17.05 s+1)}$$

$$\text{Plate 11: } \frac{x_n(s)}{f(s)} = \frac{.0039 (37.16 s+1)}{(47.66 s+1)(18.05 s+1)}$$

$$\text{Plate 13: } \frac{x_n(s)}{f(s)} = \frac{0.0019}{(47.66 s+1)}$$

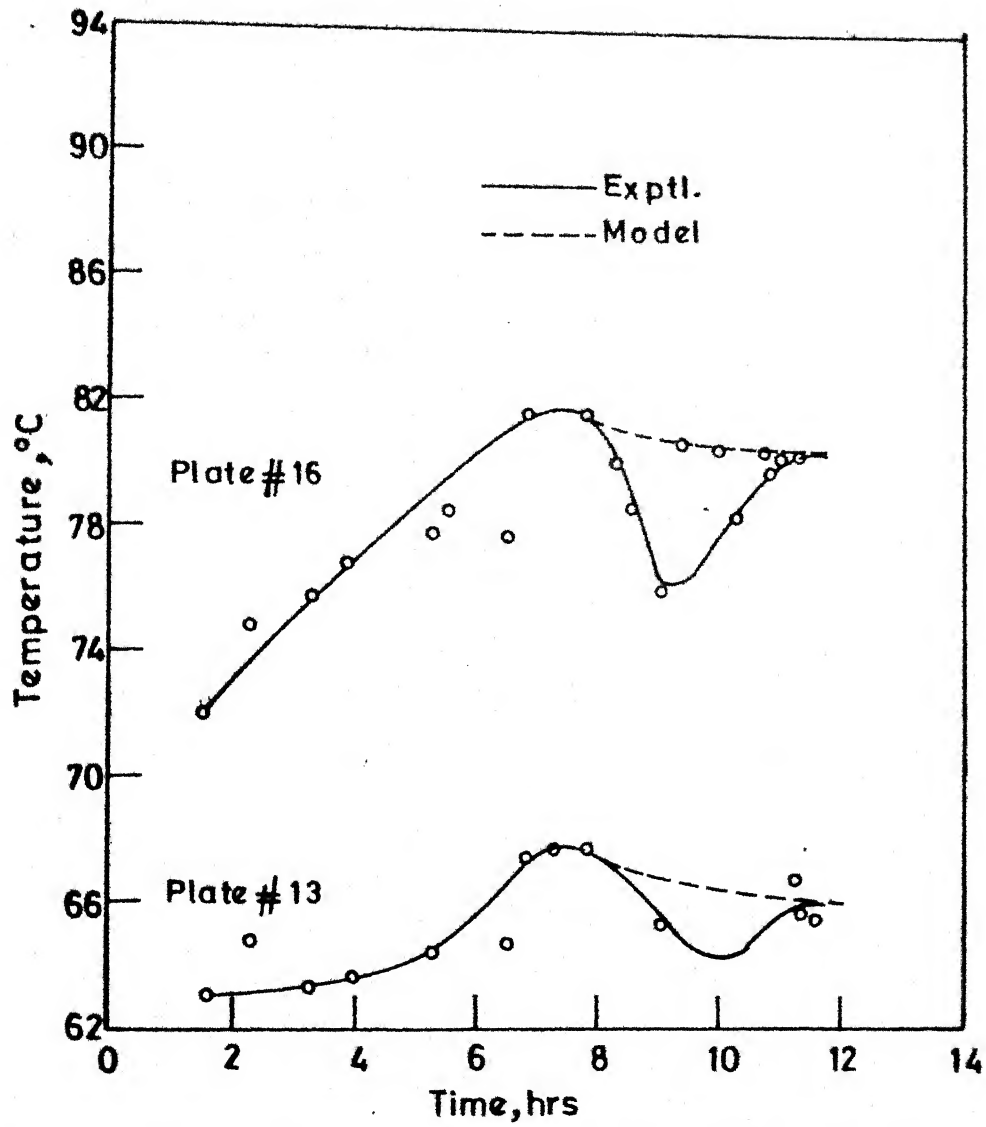


FIG. 4 Temperature response for two typical trays in stripping section for step disturbance in feed rate (Run1)

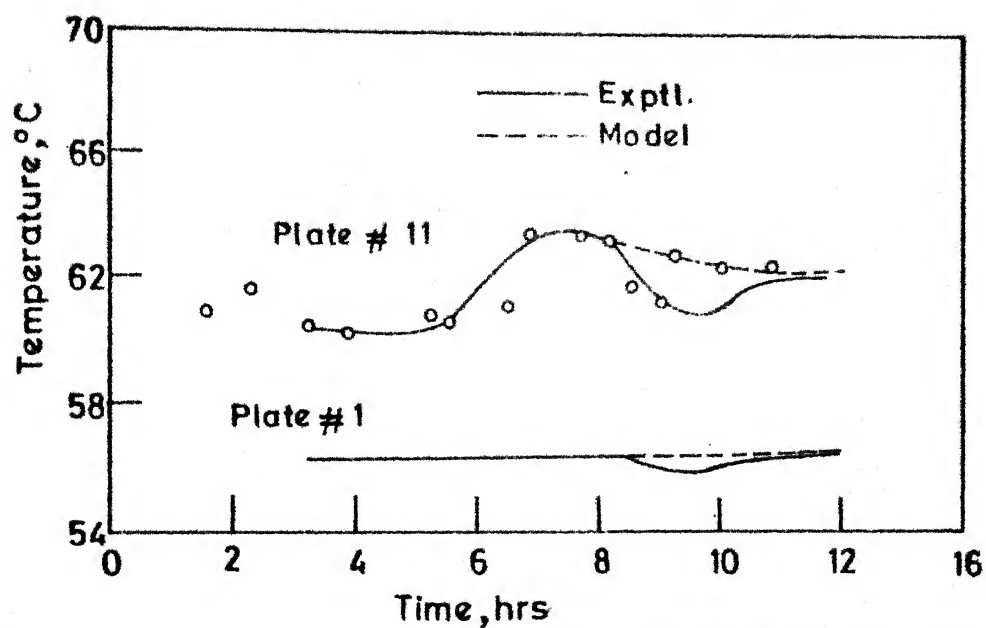


FIG. 5 Temperature response for two typical trays in enriching section for step disturbance in feed rate (Run 1)

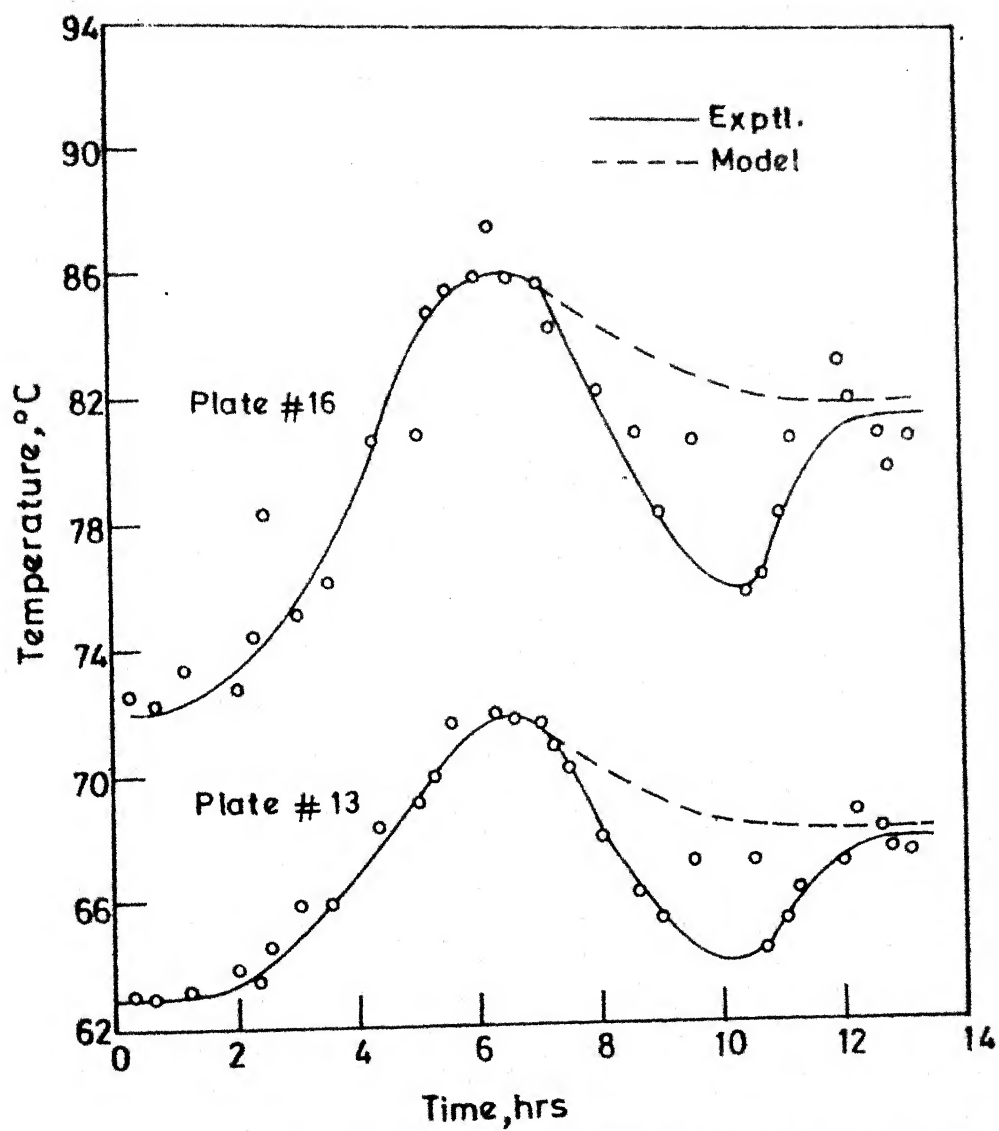


FIG 6 Temperature response for two typical trays in stripping section for step disturbance in feed rate (Run 3)

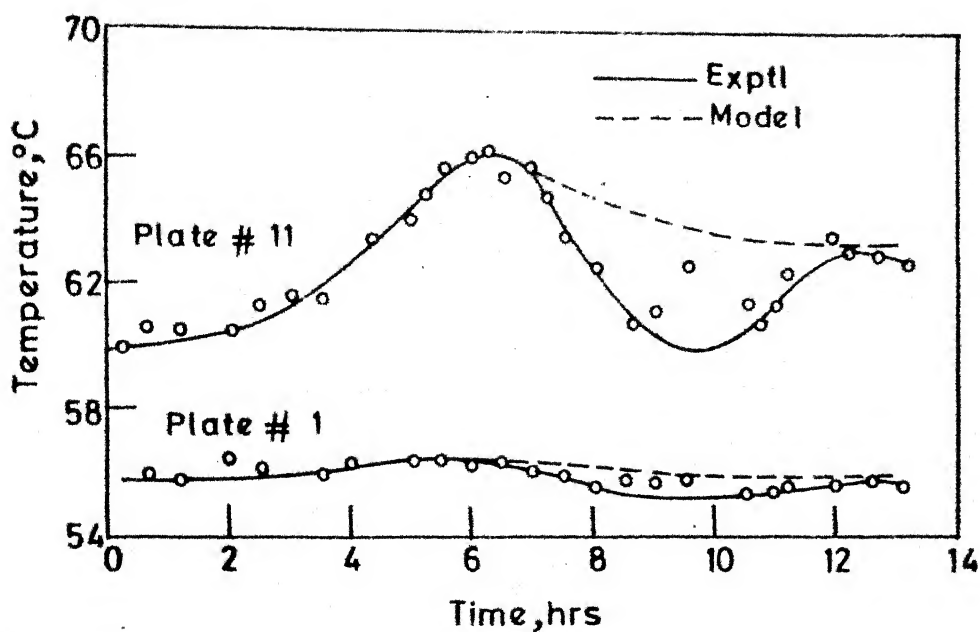


FIG. 7 Temperature response for two typical trays in enriching section for step disturbance in feed rate (Run 3)

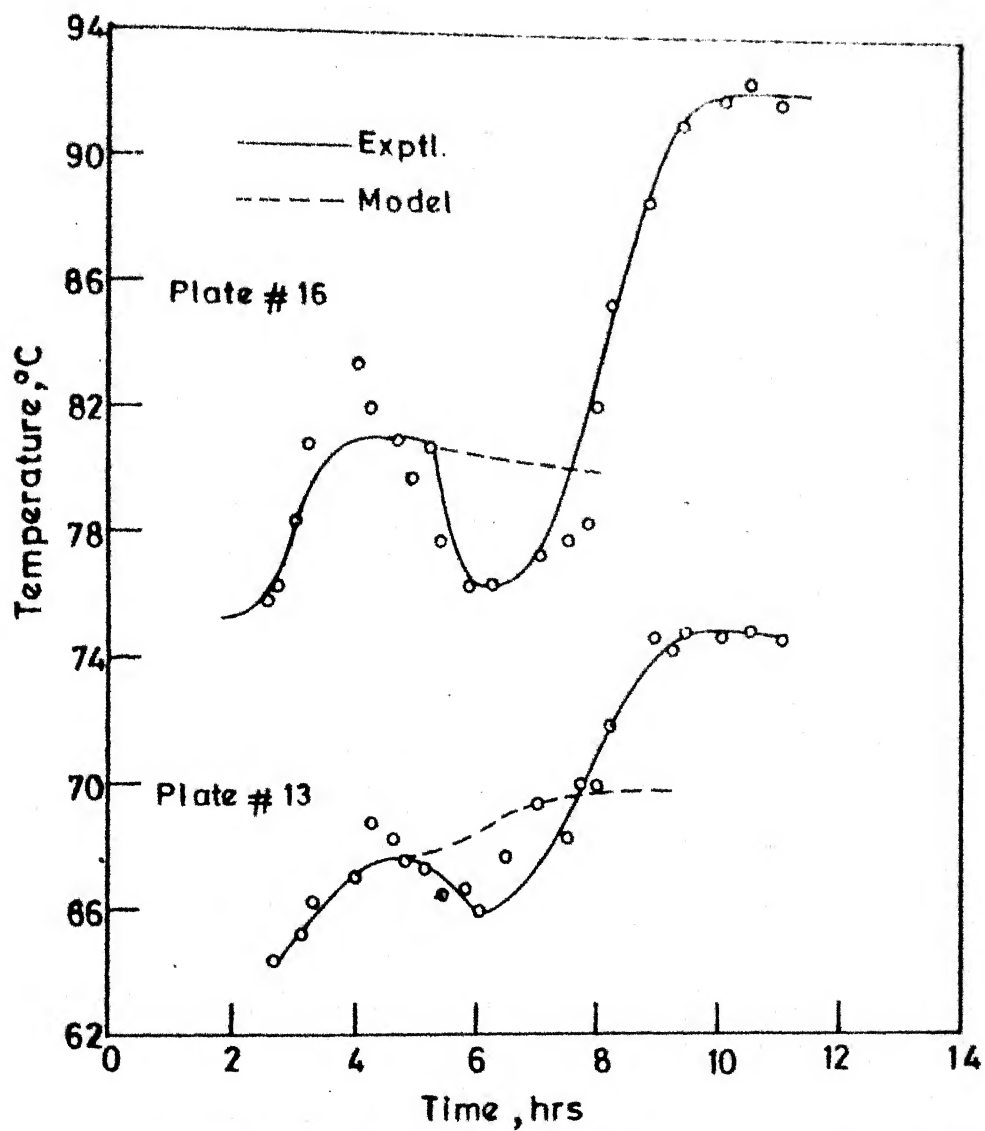


FIG. 8 Temperature response for two typical trays in stripping section for step disturbance in feed composition (Run 4)

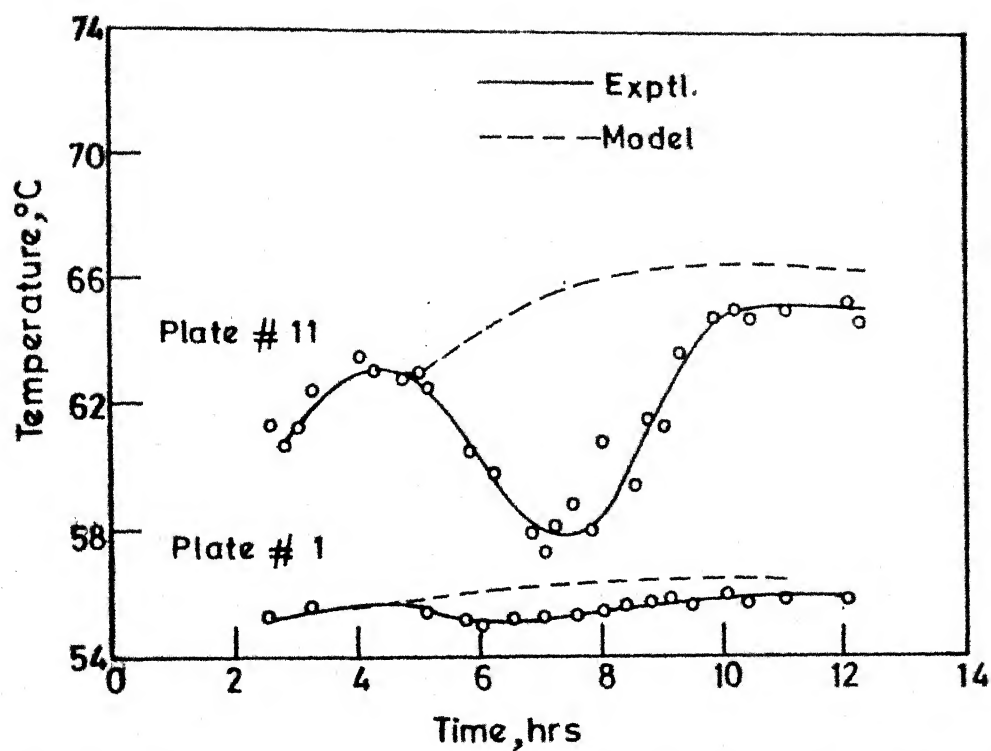


FIG. 9 Temperature response for two typical trays in enriching section for step disturbance in feed composition (Run 4)

$$\text{Plate 16: } \frac{x_n(s)}{f(s)} = \frac{0.0006}{(47.66 s+1)}$$

For feed composition load, x_F

$$\text{Plate 1: } \frac{x_n(s)}{x_F(s)} = \frac{0.9548 (1.124 s+1)}{(47.66 s+1)(12.70 s+1)}$$

$$\text{Plate 11: } \frac{x_n(s)}{x_F(s)} = \frac{0.2690 (37.16 s+1)}{(47.66 s+1)(37.15 s+1)}$$

$$\text{Plate 13: } \frac{x_n(s)}{x_F(s)} = \frac{0.0284 (-6.72 s+1)}{(47.66 s+1)(33.73 s+1)}$$

$$\text{Plate 16: } \frac{x_n(s)}{x_F(s)} = \frac{-0.0979 (-11.20 s+1)}{(+7.66 s+1)(38.38 s+1)}$$

Figures 4 to 9 show experimental values of temperature as function of time for various plates. Also plotted on the same figures are the temperatures expected on the respective trays predicted by the model.

It is seen from these figures that the predicted values agree with the experimental data only as the second steady state approaches. In between the first and second steady state, the experimental profiles show a dip which is more pronounced near the feed tray. The lack of fit during the unsteady state operation for a step change in feed flow rate (Figures 4 to 7) can be attributed to introduction of cold feed which lowers the feed flow temperature reducing vapor flow upwards and its temperature and increasing liquid flow downwards with lower temperature. This effect, however is minimal on plate 1 as expected. A similar observation was noted by Beaverstock and Harriot (1973)

even for reflux controlled column.

For step disturbance in feed composition, Figures 8 and 9 show the experimental temperatures to be higher than predicted in the stripping section. The step change of -46.6 percent is really too high to allow the assumptions to hold. In particular, the analysis requires that because of composition change no significant change in molar flow rates result. However, in the present case the vapor flow rates changed significantly (The vapor flow rate in stripping section changed by over 20% and in the enriching section it changed by over 30%) causing deviations. Because of this drastic change, there was flooding in the lower section. This resulted in the temperature of the liquid (with a lower acetone content) rising up.

The location of a sensor for a single point control should be on the tray where the gain is high. Also, the steady state error should be low. Wahl and Harriot (1971) reported that the optimum location of the sensor for top product control, for step disturbance would be in plate 1 itself.

Experimental Difficulties and Sources of Error:

1. In the existing set up gravity feed was used. However, due to the vapors in the column, there was a constant locking up of feed. This problem was partially overcome by venting the vapors from time to time from the feed line. But, this caused a disturbance in the column. Moreover, between the two successive venting of vapors from the feed line, there was a

continuous decline of feed rate and assumption of constant feed rate was **far** from satisfactory. A positive displacement pump of variable capacity should be used for the constancy of feed flow rate.

2. Feed Temperature:

In absence of a temperature controller on the feed preheater, it was not possible to maintain a constant feed temperature. Cold feed was, therefore, introduced which caused a thermal disturbance resulting in abnormal behavior as discussed above.

3. Reflux Rate and Temperature:

Unless steady state is reached, the rate of reflux would be varying making steady state still more elusive. Also if the reflux temperature is not at its bubble point then it will cause another thermal disturbance in the column. However, without a temperature control on the reflux or on the condenser it was not possible to avoid the disturbance in the present study.

4. Composition Measurement:

The compositions in the condenser, first, tenth and sixteenth plates and the reboiler were periodically checked using a refractometer. However, except for the distillate, the remaining samples showed erroneous readings. This is because of impurities present. Refractive index measurements,

being sensitive to impurities are, not reliable. It is suggested that an on-line gas chromatograph should be used along with appropriate arrangements for a pure vapor sample to be analyzed. This will enable the time delays to be measured accurately.

5. Flow Measurements:

As discussed earlier, the latex rubber sock, separating the silicone oil filled in the DP cell from the working fluid, tended to rupture making the measurements of differential pressure across the orifice plates inaccurate. Moreover, the present DP cells were found to be very sensitive to temperature and mechanical vibrations.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

While the present study provided an open loop dynamic model for the distillation column, there were several shortcomings in the experimental program. This being the first investigation on the dynamics of a pilot plant scale multi-stage column in this laboratory, it was mostly exploratory in nature. Successive investigations should result in more exact dynamic studies after the shortcomings reported in this work have been overcome. With some control on reflux flow and its temperature and on feed flow and its temperature and data logging facility on microprocessor it would be possible to develop better open loop as well as closed loop dynamic models for the column. More sophisticated multiple variable, multiple time delay models, which could be easily developed after providing some distributed control as suggested above, will enable designing overall control strategy for the column.

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APPENDIX 1A.1.1 Derivation of Unsteady State Equations for All The Loads and Simplified Model Time Constant:

(a) Feed composition load

The unsteady state component balance with the perturbation is (Figure 1)

$$F(X_F + x_F) = D(X_O + x_O) + B(X_R + x_R) + H_T \dot{x}_{av} \quad (A1.1.1)$$

The steady state component balance is

$$FX_F = DX_O + BX_R \quad (A1.1.2)$$

substituting Eq. A1.1.2 in Eq. A1.1.1 we get

$$H_T \dot{x}_{av} = Fx_F - Dx_O + Bx_R \quad (A1.1.3)$$

where $\dot{x}_{av} = dx_{av}/dt$

(b) Feed rate load

The unsteady state component balance for feed rate load is

$$H_T \dot{x}_{av} = FX_F + fX_F - DX_O - Dx_O + BX_R + Bx_R + fX_R + fx_R \quad (A1.1.4)$$

Substituting A1.1.2 in A1.1.4 we get

$$H_T \dot{x}_{av} = f(X_F - X_R) - Dx_O - Bx_R \quad (A1.1.5)$$

The composition x_{av} , will completely define the composition x , through the column. In this case

$$x_O = f_1(x_{av}) \quad (A1.1.6)$$

$$x_R = f_2(x_{av}) \quad (A1.1.7)$$

Linearizing Eqns. A1.1.6 and A1.1.7 we get

$$x_o = C_o x_{av} \quad (\text{A1.1.8})$$

$$x_R = C_R x_{av} \quad (\text{A1.1.9})$$

where

$$C_o = \frac{dx_o}{dx_{av}} \quad \text{and} \quad C_R = \frac{dx_R}{dx_{av}}$$

Equations A1.1.3 and A1.1.5 can be rearranged to give

$$T_s \left(\frac{dx_{av}}{dt} \right) + x_{av} = \frac{\text{Load}}{BC_R + DC_o} \quad (\text{A1.1.10})$$

where

$$T_s = \frac{H_T}{(BC_R + DC_o)}$$

$$\text{Load} = Fx_F \text{ or } X_F f$$

C_o may be evaluated as follows:

$$\begin{aligned} C_o &= \frac{dx_o}{dx_{av}} = \left(\frac{\delta x_o}{u} \right) \left(\frac{\delta u}{x_{av}} \right) \\ &= \frac{G_{ou}}{(\delta x_{av} / \delta u)} \end{aligned} \quad (\text{A1.1.11})$$

where G_{ou} = Gain of the condenser due to load u

u = load f , or x_F

C_R can also be found similarly.

Also

$$\frac{\delta x_{av}}{\delta u} = \sum_{n=0}^{N+1} \frac{H_n G_{nu}}{H_T}$$

where

G_{nu} = Gain on plate n due to load u .

Substituting Eqs. A1.1.11 and A1.1.12 the equations for C_o and C_R become

$$C_o = H_T G_{ou} / \sum_{n=0}^{N+1} H_n G_{nu} \quad (A1.1.13)$$

$$C_R = H_T G_{Ru} / \sum_{n=0}^{N+1} H_n G_{nu} \quad (A1.1.14)$$

Substituting the value of C_o and C_R in the definition of T_s , we get

$$T_s = \frac{\sum_{n=0}^{N+1} H_n G_{nu}}{B G_{Ru} + D G_{ou}} \quad (A1.1.15)$$

where

n = plate number, 0 is condenser, $N+1$ is reboiler

H_n = holdup for plate n .

A1.2 Derivation of Constants in Wahl and Harriots Model:

The constants a_n and b_n are presented in Table 1 in Chapter 2. This derivation has been necessiated because of some misprints in the original paper (Wahl and Harriot, 1971).

for $n = 0$ $b_n = 1$ and $a_n = 0$

for $n = 1$ $b_n = 1/K_1$ and $a_n = 0$

for $n = 2$

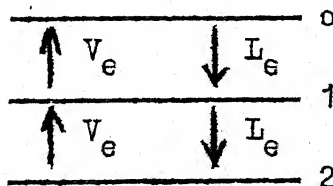


Figure: A1.1

A component balance around tray 1 (Figure A1.1) for perturbation in composition gives

$$V_e y_2 - V_e y_1 + L_e x_0 - L_e x_1 = 0 \quad (A1.2.1)$$

The equilibrium relation ship is

$$K_n = \frac{y_n}{x_n} \quad (A1.2.2)$$

where

$$y_n = y_n^{(2)} - y_n^{(1)}$$

$$x_n = x_n^{(2)} - x_n^{(1)}$$

$x_n^{(1)}$, $x_n^{(2)}$ are liquid compositions on tray n at first and second steady state respectively.

$y_n^{(1)}$, $y_n^{(2)}$ are vapor compositions on tray n at first and second steady state respectively.

Substituting A1.2.2 in A1.2.1 and rearranging we get for a load disturbance, u

$$\left(\frac{x_2}{u}\right) = \frac{\left[V_e K_1 + L_e\right] \left(\frac{x_1}{u}\right) - L_e \left(\frac{x_0}{u}\right)}{V_e K_2}$$

but $\frac{x_1}{u} = \frac{(x_0/u)}{K_1}$

Therefore

$$\begin{aligned} \left(\frac{x_2}{u}\right) &= \frac{\left(\frac{V_e K_1 + L_e}{K_1}\right) \left(\frac{x_0}{u}\right) - L_e \left(\frac{x_0}{u}\right)}{V_e K_2} \\ &= \left[\left(\frac{V_e K_1 + L_e}{V_e K_2}\right) \frac{1}{K_1} - \frac{L_e}{V_e K_2} \right] \left(\frac{x_0}{u}\right) \end{aligned}$$

$$\text{i.e. } b_2 = \left[\frac{V_e K_1 + L_e}{V_e K_2} \right] b_1 - \frac{L_e}{V_e K_2} b_0$$

$$\mathcal{E} = 0$$

similarly for trays 3, ..., N_F we get

$$b_n = \frac{V_e K_{n-1} + L_e}{V_e K_n} b_{n-1} - \frac{L_e}{V_e K_n} b_{n-2} \quad (\text{A1.2.3})$$

$$\mathcal{E} = 0$$

For tray $n = n_{F+1}$

A component balance around feed tray (Fig. A1.2) and rearrangement gives

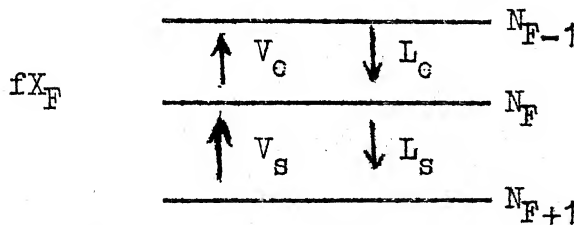


Figure A1.2

$$\left(\frac{x_n}{u} \right) = \left[\frac{V_e K_{n-1} + L_s}{K_n V_s} \right] b_n - \frac{L_e}{K_n V_s} b_{n-1} + \frac{x_{n-1} - x_F}{K_n V_s}$$

i.e.

$$b_n = \left(\frac{V_e K_{n-1} + L_s}{K_n V_s} \right) b_{n-1} - \frac{L_e}{K_n V_s} b_{n-2} \quad (\text{A1.2.4})$$

$$a_n = \frac{x_{n-1} - x_F}{K_n V_s} \quad (\text{A1.2.5})$$

$$\mathcal{E} = 0$$

Assuming that the load is transmitted instantly in the flow response, the constants for $n = N_{F+2}$ to N_R can be found as in enriching section. They are as follows:

$$b_n = \left(\frac{V_s K_{n-1} + L_s}{V_s K_n} \right) b_{n-1} - \frac{L_s}{V_s K_n} b_{n-2} \quad (A1.2.6)$$

$$F = \left(\frac{V_s K_{n-1} + L_s}{V_s K_n} \right) a_{n-1} - \frac{L_s}{V_s K_n} a_{n-2} \quad (A1.2.7)$$

$$a_n = F + \frac{X_{n-1} - X_F}{V_s K_n} \quad (A1.2.8)$$

The successive substitution of the solution of $\left(\frac{x_n}{u}\right)$ in the subsequent stage starting from tray 1 yields

$$\left(\frac{x_n}{u}\right) = a_n + b_n \left(\frac{x_o}{u}\right) \quad (A1.2.9)$$

where $\left(\frac{x_o}{u}\right)$ is the condenser gains G_{ou} .

The condenser gain G_{ou} is found as follows:

a) Feed Composition load

The overall steady state component balance for the perturbed column is

$$B(X_R + x_R) + D(X_o + x_o) = F(X_F + x_F) \quad (A1.2.10)$$

Before the column is perturbed the component balance is

$$DX_o + B X_B = F X_F \quad (A1.2.11)$$

Substituting Eq. A1.2.11 in A1.2.10 we get

$$D\left(\frac{x_o}{x_F}\right) = F - B \left(\frac{x_R}{u}\right) \quad (A1.2.12)$$

Solving Eq. A1.2.9 and Eq. A1.2.12 simultaneously for x_o/x_F we get

$$\frac{x_o}{x_F} = \frac{F - D a_R}{D + B b_R} \quad (A1.2.13)$$

The gain for each plate is calculated using Eqns. A1.2.9 and A1.2.13.

(b) For feed rate load

The overall component balance for perturbed column is

$$(F+f) X_F = D(X_O+x_O) + (B+f) (X_B+x_B) \quad (A1.2.14)$$

Substituting eq.A1.2.11 in Eq.A1.2.14 and solving simultaneously with Eq.A1.2.12 we get

$$\left(\frac{x_O}{F}\right) = \frac{X_F - \bar{X}_R - Ba_R}{D + F_R}$$

The condenser gain for reflux rate load and boilup loads can be similarly found.

APPENDIX 2

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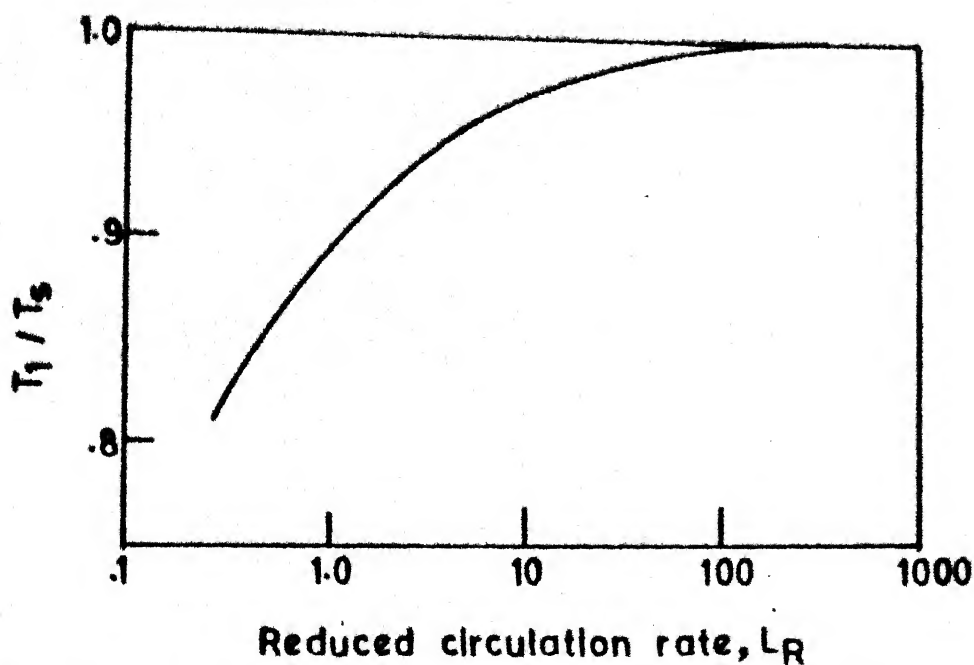


FIG. A.2.1 Prediction of principal time constant

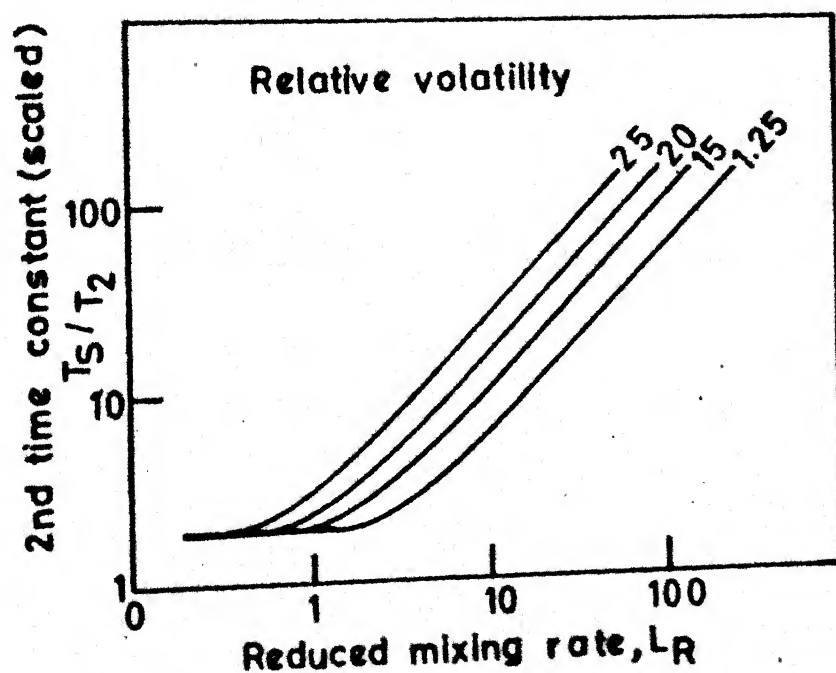


FIG. A.2.2 Prediction of second time constant

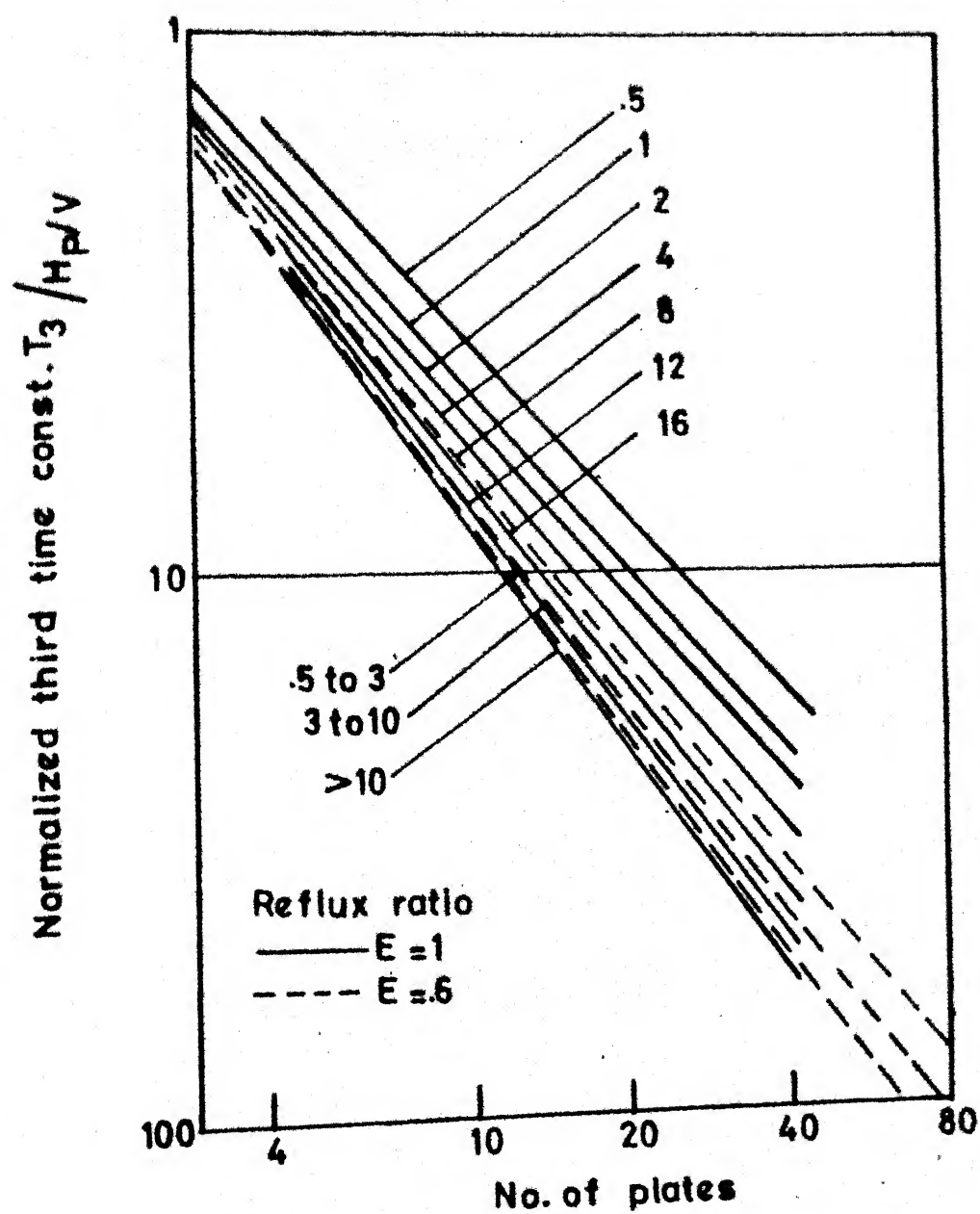


FIG. A 2.3 Prediction of third time constant.

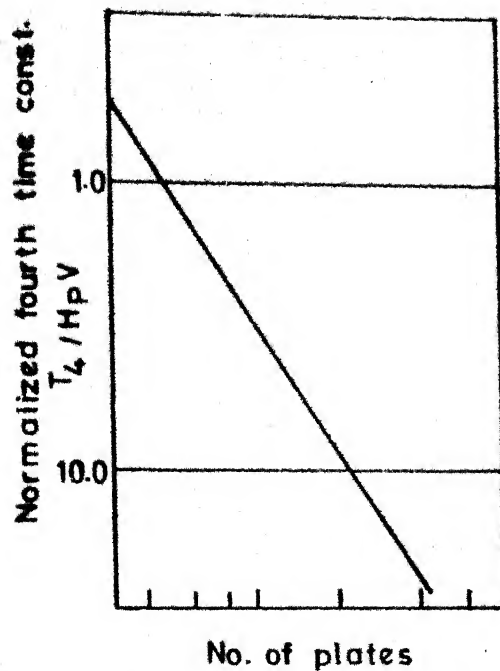


FIG.A.2.4 Prediction of fourth time constant

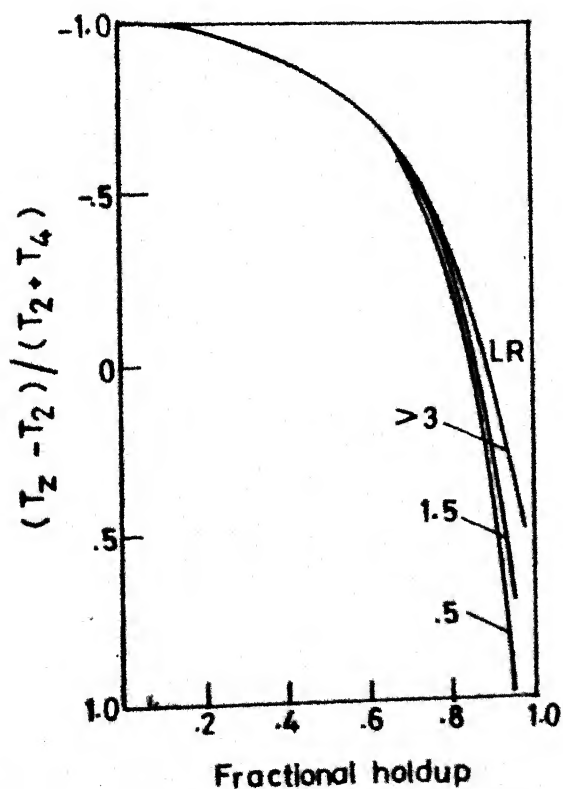


Fig. A.2.5 Prediction of reciprocal zero T_z

APPENDIX 3

A3.1 Determination of Reflux Ratio Required for Desired Separation of Acetone-Water Mixture Feed:

Feed

Mole fraction acetone, $X_{AF} = 0.15$

Mole fraction water, $X_{WF} = 0.85$

Distillate:

Mole fraction acetone, desired

$$X_{A,D} = 0.965$$

Mole fraction water, desired,

$$X_{W,D} = 0.035$$

Bottoms

Mole fraction acetone, desired,

$$X_{A,B} = 0.02$$

Mole fraction water, desired,

$$X_{W,B} = 0.98$$

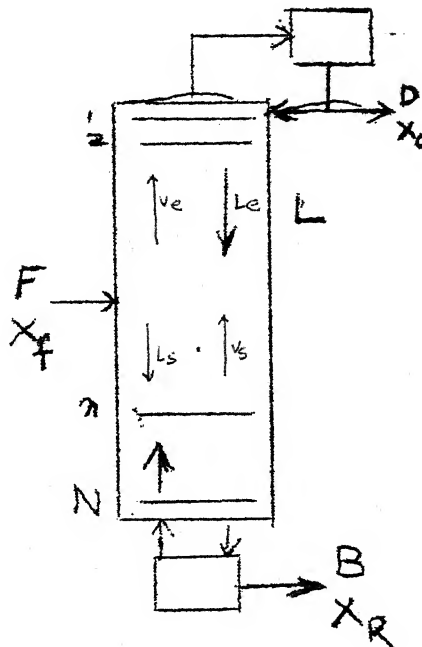


Figure: A3.1

Let F be the molar flow rate of Feed, D is the top product rate,

B is the bottoms rate

Then mass balance equations are

(i) Overall mass balance:

$$F = D + B$$

(ii) Overall component balance, acetone

$$F X_{A,F} = D X_{A,D} + B X_{A,B}$$

(A3.1.2)

On solving A3.1 and A3.2 simultaneously we get

$$D = 0.138 F \quad \text{and} \quad B = 0.862F$$

For a feed rate $F = 35.38 \text{ gmole/min}$,

$$D = 4.882 \text{ gmole/min}$$

$$B = 30.513 \text{ gmole/min.}$$

McCabe - Thiele procedure has been used to find the suitable reflux ratio by trial and error (Treybal, 1980). The operating line for the enriching section has a slope of $R/R+1$.

For finding the operating line of the stripping section, the q line is first plotted. The ' q ' line is found as follows:

Average molecular weight of feed (M_{av}) is

$$\begin{aligned} M_{av} &= 0.15 \times 58.08 + 0.85 \times 18.016 \\ &= 24.026 \end{aligned}$$

For an acetone solution in water of 0.15 mole fraction, the saturation temperature

$$T_{sat} = 63.5^\circ\text{C}$$

The molar heat of solution $H_s = -338 \text{ Btu/lb.mol} = -187.38 \text{ cal/gmole}$ at 15°C

For a feed temperature of 25°C

$$C_{p, \text{ water}} = 1 \text{ Btu/lb}^\circ\text{F} = 1 \text{ Cal/gms } ^\circ\text{C}$$

$$C_{p, \text{ acetone}} = 0.53 \text{ Btu/lb } ^\circ\text{F} = 0.53 \text{ cal/gm}^\circ\text{C}$$

Let the base temperature be 15°C .

Then molar enthalpy of feed H_f is

$$\begin{aligned} H_f &= M_w [X_{w,F} C_{p,w} (T - T_{base})] + M_A [X_{A,F} C_{p,A} (T - T_{base})] + H_s \\ &= 18.016 [.85 \times 1 \times (25 - 15)] + 58.08 [.15 \times 0.53 \times (25 - 15)] - 187.38 \\ &= 11.507 \text{ cal/gmole} \end{aligned}$$

Similarly the molar enthalpy of saturated liquid

$$H_L = 1409.578 \text{ Btu/lb mole} = 781.47 \text{ cal/gmole}$$

Latent heat of vaporization of acetone at 63.5°C is

$$H_{s,A} = 7244.89 \text{ Cal/gmole}$$

Latent heat of vaporization of water at 63.5°C

$$H_{s,W} = 9988 \text{ cal/gmole}$$

The molar enthalpy of saturated vapor at 63.5°C is

$$H_G = H_L + X_{A,F} H_{s,A} + X_{W,f} H_{s,W}$$

$$= 8437.84 \text{ cal/gmole}$$

$$q = \frac{H_G - H_f}{H_G - H_L} = \frac{8437.84 - 11.507}{8437.84 - 781.47} = 1.1$$

The data has been taken from (Perry, 1973).

Therefore slope of q line $= \frac{\bar{q}}{q-1} = \frac{1.1}{0.1} = 11$

Knowing the q line, the stripping section operating line can be drawn. Once the operating lines are known the number of stages can be found. This procedure is repeated for 2 values of R .

(i) $R=1$

Number of stages in enriching section = 13

Number of stages in stripping section = 2

Total number of ideal stages = 15

Taking an overall efficiency of 0.75, the real trays needed will be $\frac{15}{.75} = 20$

Since the total number of actual stages is 16,

$R = 1$ is insufficient.

(ii) $R = 2.5$

Number of stages in enriching section = 6

Number of stages in stripping section = 2

Total number of ideal stages = 8

With a stage efficiency of $E = 0.75$ this is sufficient. The equilibrium curve and the stage determination are shown in Figures A3.2 and A3.3. A pseudo equilibrium curve has been drawn (Treybal, 1980), and the number of trays found using this. The temperature versus X-Y is given in Figure A3.4. The details of column design are given in Saboo and Sivaram (1980) and Mistry (1974).

A3.2 Determination of Liquid and Vapor Flow Rates in Stripping and Enriching Sections and the Column Capacity:

The procedure is given in Treybal (1980)

From Appendix A3.1

$$R = 2.5$$

$$D = 0.138 F$$

$$B = 0.862 F$$

$$\text{For } F = 35.38 \text{ gmole/min,}$$

$$D = 4.882 \text{ gmole/min}$$

$$B = 30.513 \text{ gmole/min}$$

$$V_e = (R + 1) D$$

$$= 15.613 \text{ gmole/min}$$

$$L_e = RD = 10.730 \text{ gmole/min.}$$

$$V_s = V_e + F (q-1)$$

$$= 19.151 \text{ gmole/min}$$

$$L_s = F + L_e + V_s - V_e$$

$$= 49.648 \text{ gmole/min}$$

The maximum permissible flow rate for various sections of the column are as follows:

At the top: 183.8 kg/hr

At the feed tray: 176.4 kg/hr

At the bottom: 221.04 kg/hr

The detailed calculations are shown in Saboo and Sivaram (1980).

Determination of Reboiler Duty:

Rate of vapors generated $V_s = 19.151 \text{ gmole/min}$

Latent heat of vaporization $H_L = 9720 \text{ cal/gmole}$

$$\text{Heat duty, } Q_R = 9720 \times 19.151$$

$$= 186138 \text{ cal/min}$$

$$= 738.6 \text{ Btu/min}$$

Latent heat of steam at 30 psig $H_{Ls} = 1164.1 \text{ Btu/lb}$

$$\text{Therefore, steam required} = \frac{738.6}{1164.1}$$

$$= .634 \text{ lb/min} = .288 \text{ kg/min}$$

Determination of Condenser Duty:

For a maximum flow rate of 150 kg/hr Saboo et al. (1980) have shown the calculations for condenser duty.

The required heat transfer coefficient U was found to be $U = 55.97 \text{ cal/hr. cm}^2 \text{ }^\circ\text{C}$

The designed U for the condenser (Mistry, 1974), is $70.30 \text{ cal/hr.cm}^2 \text{ }^\circ\text{C}$. Therefore, the condenser which is used for a cold reflux is sufficient as the flow rate used ($F = 35.38 \text{ gmole/min}$) is much below the maximum permissible flow rate.

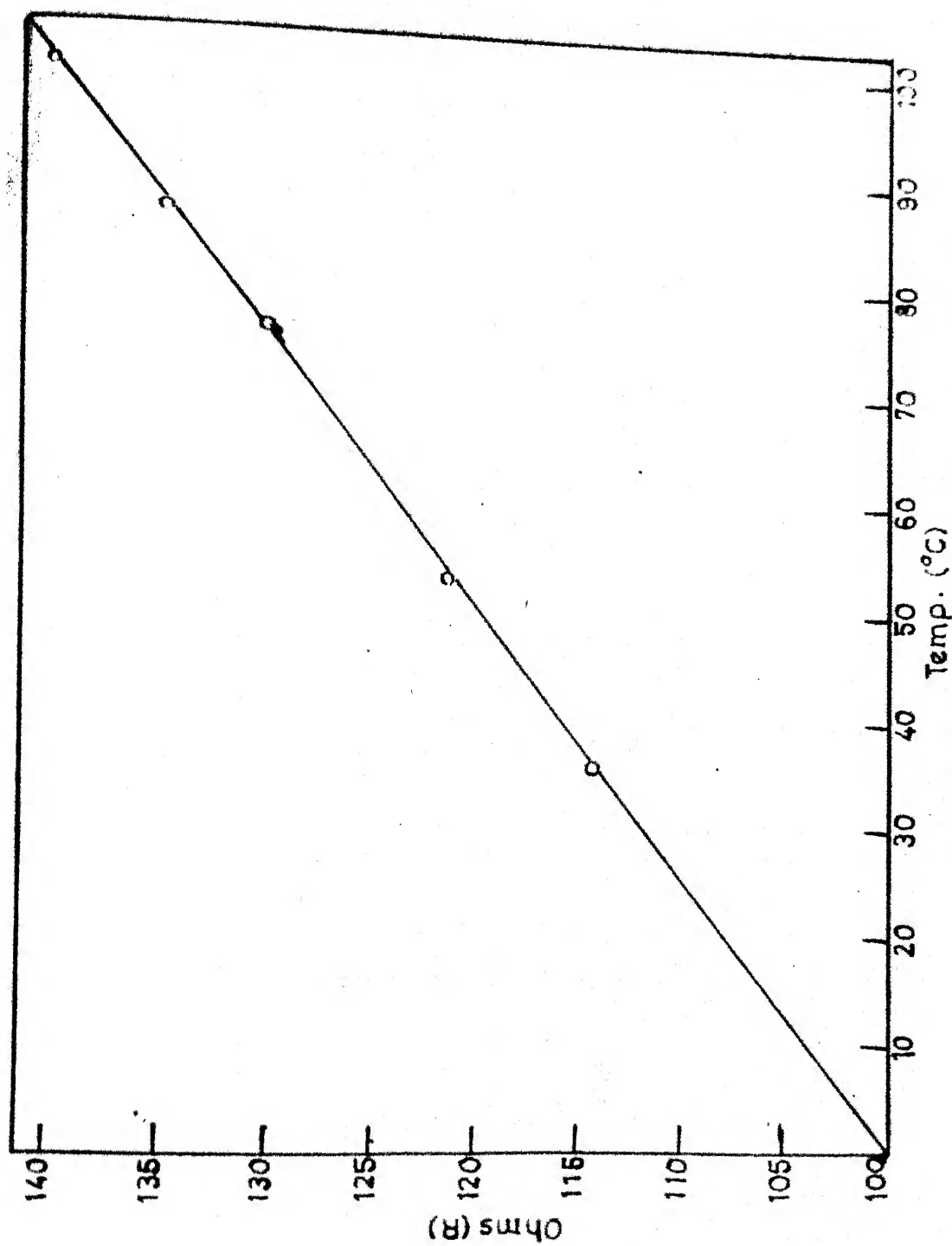


FIGURE A3.1: Temperature vs resistance graph for platinum resistance thermometer

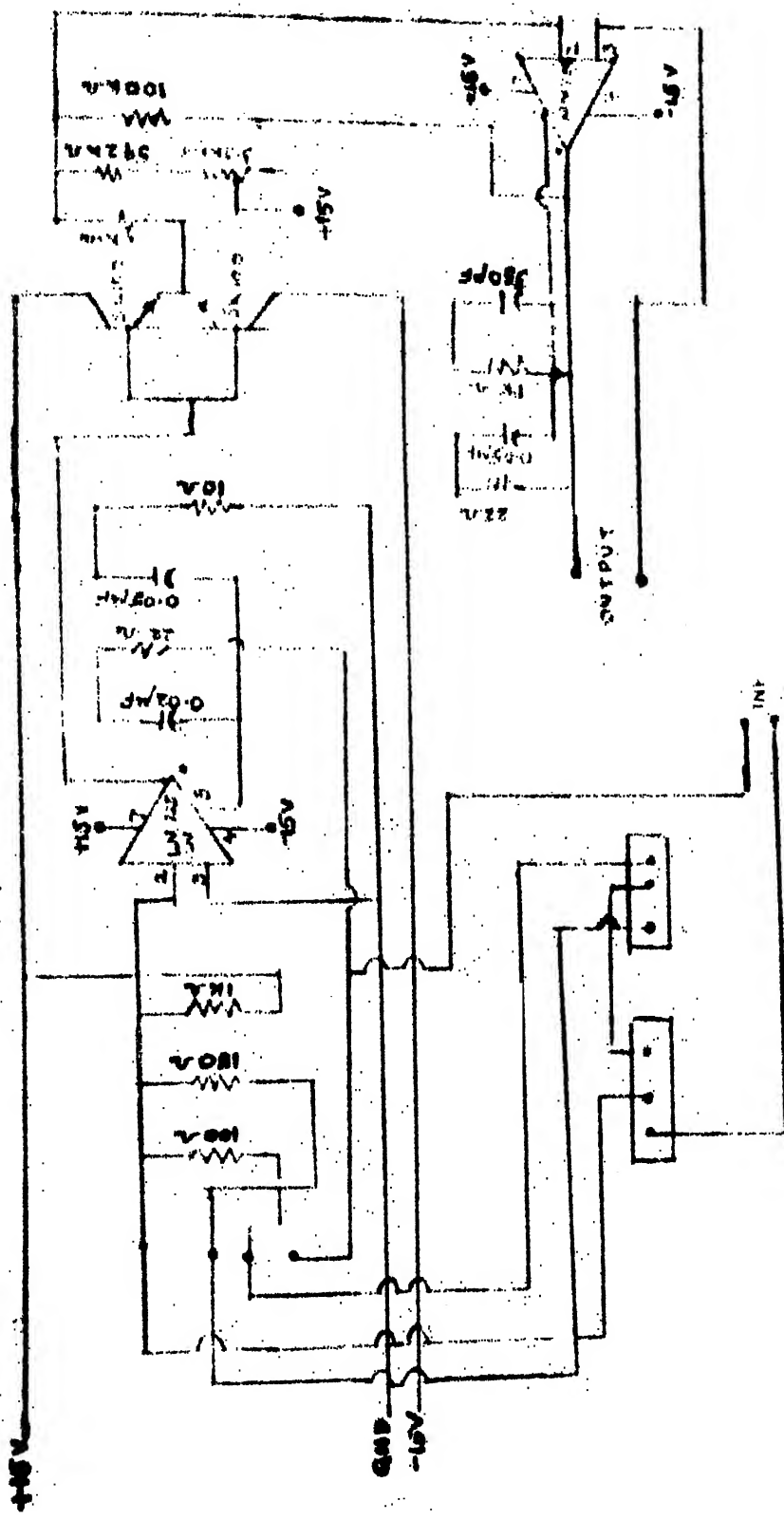


Figure A3.2: Voltage conversion circuit

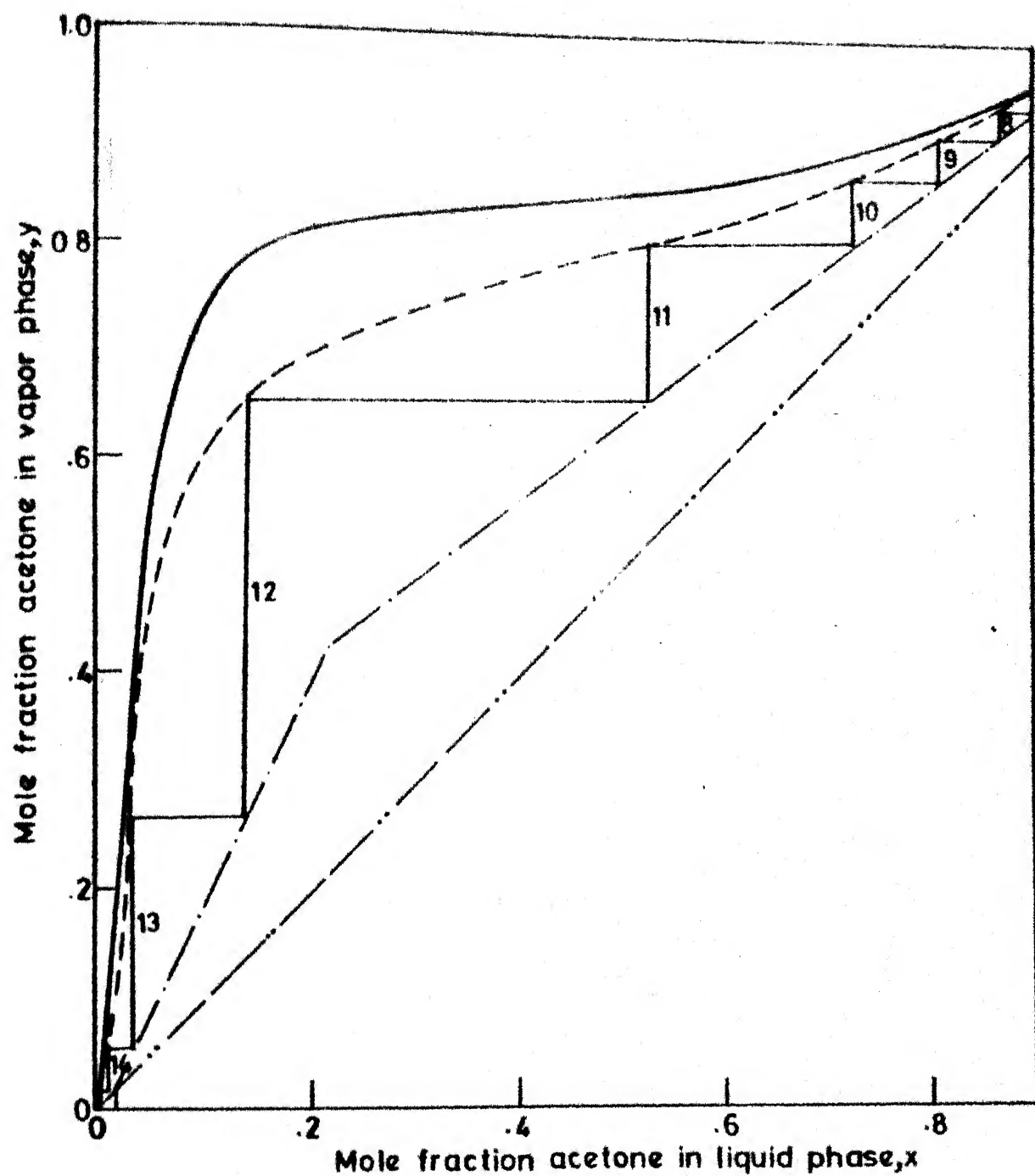


FIG. A.32 McCabe Thiele diagram for acetone - water system

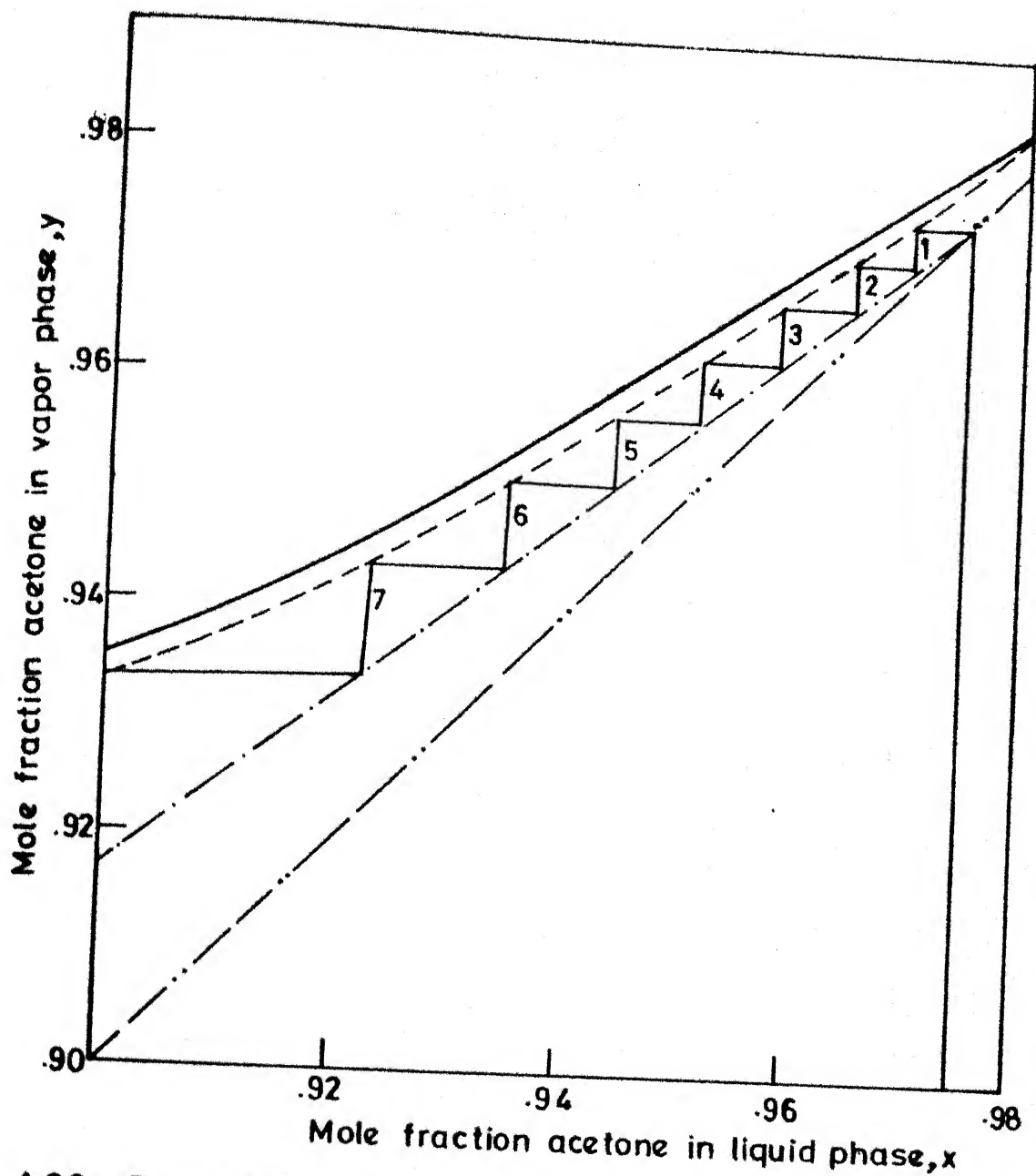


FIG. A.33 Expanded McCabe Thiele diagram for acetone-water system

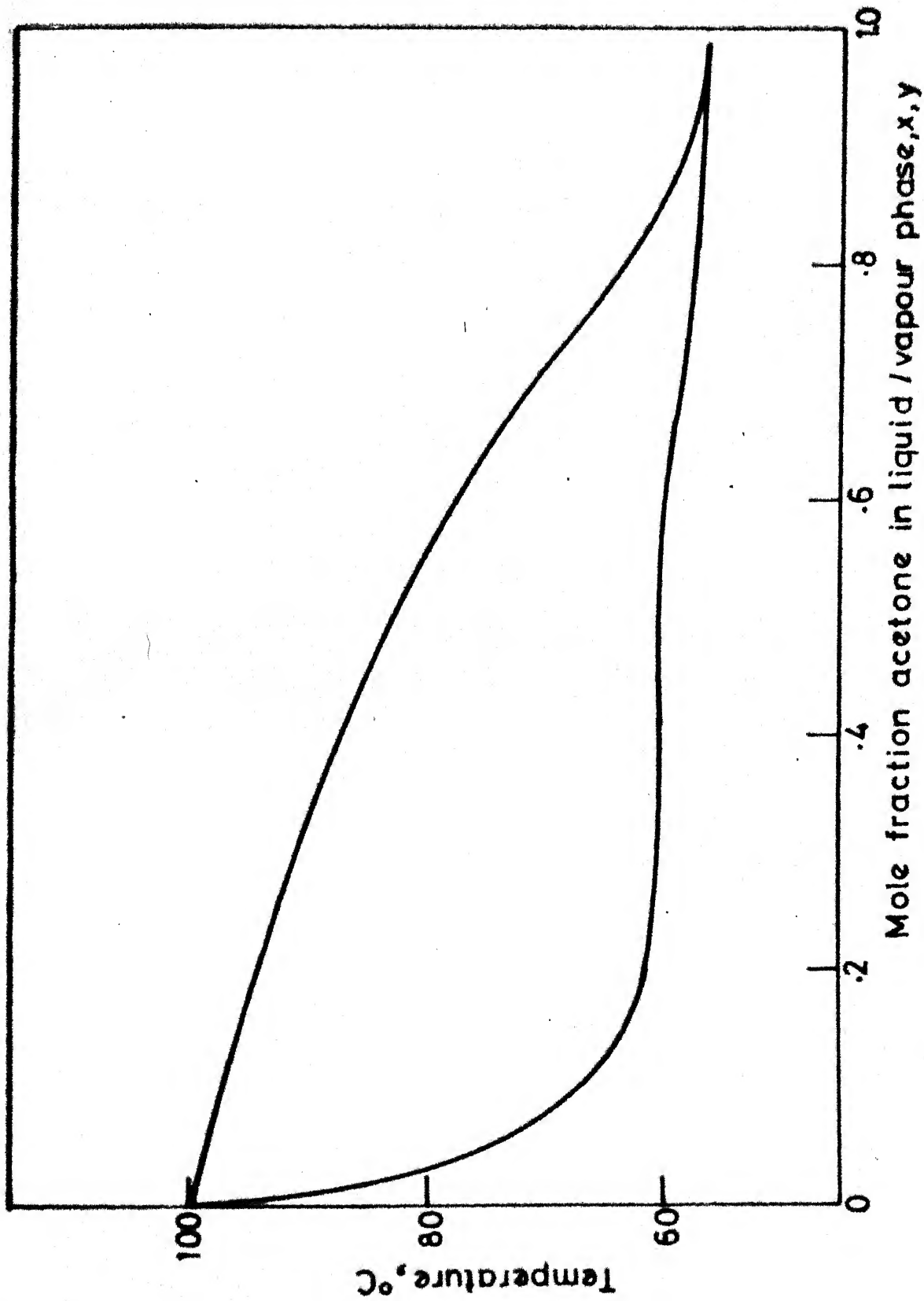


FIG. A.3.4 Temperature vs x,y diagram for acetone water system
(Othmer D.F. and Benanati R.F., Ind. Eng. chem. 37, (1945) p.299